TOWARD LOWER ORGANIC ENVIRONMENTS IN ASTROMATERIAL SAMPLE CURATION FOR DIVERSE COLLECTIONS.

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Introduction: Great interest was taken during the frenzied pace of the Apollo lunar sample return to achieve and monitor organic cleanliness. Yet, the first mission resulted in higher organic contamination to samples than desired. But improvements were accomplished by Apollo 12 [1]. Quarantine complicated the goal of achieving organic cleanliness by requiring negative pressure glovebox containment environments, proximity of animal, plant and microbial organic sources, and use of organic sterilants in protocols. A special low organic laboratory was set up at University of California Berkeley (UCB) to cleanly subdivide a subset of samples [2, 3, 4]. Nevertheless, the basic approach of handling rocks and regolith inside of a positive pressure stainless steel glovebox and restricting the tool and container materials allowed in the gloveboxes was established by the last Apollo sample return. In the last 40 years, the collections have grown to encompass Antarctic meteorites, Cosmic Dust, Genesis solar wind, Stardust comet grains and Hayabusa asteroid grains. Each of these collections have unique curation requirements for organic contamination monitoring and control. Here is described some changes allowed by improved technology or driven by changes in environmental regulations and economy, concluding with comments on organic witness wafers. Future sample return missions (OSIRIS-Rex; Mars; comets) will require extremely low levels of organic contamination in spacecraft collection and thus similarly low levels in curation. JSC Curation is undertaking a program to document organic baseline levels in current operations and devise ways to reduce those levels.

Cleaning with Ultrapure Water: Degreasing tools and containers with Freon 113 was very effective at removing organic contaminants while leaving little hydrocarbon residue. The change to cleaning with ultrapure water (UPW) in the mid-1990s was driven by the phase out of Freon use, and the need to eliminate flammable solvents as replacements. With ionic concentrations in the very low parts per trillion and total oxidizable carbon (TOC) under 5 ppb, the UPW is an active, clean solvent. UPW is produced at 8 gal/min in a constantly flowing system from which curation cleaning facilities can tap. Tool cleaning is performed in constantly flowing streams energized ultrasonically or megasonically. For example, final rinse water analysis for flushing a stainless steel glovebox with about 50 gallons of heated UPW showed no particles >1μm size per liter, TOC of 40 ppb, only cation detectable was Fe (0.16 ppb) and only detectable anions, all <1ppb, were F, Cl, SO4, PO4. Very low particle tools and containers can be produced [5]. What is now needed is an adequately sensitive method for routine verification of surface organic cleanliness.

Cleaner nitrogen: Pure nitrogen has always been supplied to the curation gloveboxes and sample storage desiccators (which now number 43 gloveboxes and 77 storage dessicators) from the boil off of liquid nitrogen, grade C which is lower in argon (Table 1). About 1000 tons of liquid nitrogen are used annually. In a few select nitrogen-filled enclosures, point-of-use gas purifier/filters are in use. These devices produce nitrogen with < 1 ppb H_2O, O_2, CO_2, CO and retain particles > 3 nm. These devices are expensive and their long-term effects need to be evaluated.

More efficient airborne particle distribution-counting: In Apollo days environmental airborne particle counting was performed with witness plates. Hand-held airborne particle counters are now available and used to monitor all curation laboratories weekly, as well as test the integrity of HEPA and ULPA filters.

Airborne molecular contamination (AMC) monitoring: Periodic monitoring of airborne molecular contamination has been performed in Genesis laboratory since 1998 using polished silicon wafers. This need arose for laboratories in which samples were handled directly in air instead of enclosed nitrogen-filled gloveboxes. This information is especially important for laboratories that are particle-filtered with HEPA or UPLA filtration, as these filters offgas RTV compounds. For example, Genesis laboratory, with 54 ULPA and HEPA fan filter units supplied by a HEPA filtered air handler, deposits 10 ng/cm^2 on a 24-hour witness wafer. The composition is mostly siloxanes from the RTV and plasticizers. This technique captures the higher molecular weight species, likely to "stick" to sample surfaces. Molecular contamination in laboratories and gloveboxes has also been measured using sorbents, which can capture more volatile species. The polished wafer protocol has been used inside gloveboxes to assess the glovebox nitrogen environment and offgasing during heat sealing of sample bags. [6,7].

Variety in Containers: Initial organic free containers were developed at UCB from stainless steel vacuum flanges. Early low organic containers for use

in curation processing were made from drawn stainless steel with Teflon snap caps (Fig. 1). Most recently, a glass slide sandwich is used for containing Stardust and Hayabusa samples (Fig. 2). The challenges for development of suitable organic-free containers for fragile samples involve replacements for plastics used to make seals or as dunnage to prevent breakage during sample shipment.

Variety in Witness Plates: Early Apollo organic witness materials included woven aluminum mesh Fig. 3), some of which made the round trip to the Moon, and Ottowa sand, used in glovebox processing simulations. Both of these witness materials were easily subdivided for distribution. Sandford *et al*., in summarizing organic contamination in Stardust samples, recommend a variety of witness materials which are easily subdivided for distribution [8]. Some coupons saved for reference for Genesis and Stardust were not easly subdivided. Alternate witness materials suggested for laboratory reference include sapphire and CVD diamond. Need for increased availability of witness materials is suggested by the rapid accumulation of contamination acquired during storage [9]. Witness materials with a long shelf life are desired, so storage environment needs to be part of the discussion.

Fig. 1 On left is a drawn stainless container with Teflon snap cap currently used in JSC curation for rock and regolith (lunar and meteorite). On right is UCB low organic container fabricated from stainless steel vacuum flange (3 pieces). Scale is in cm.

Fig. 2. Small Stardust and Hayabusa grains are cleanly contained when placed in a dimpled glass slide sandwich.

Fig. 3. Woven aluminum mesh used as organic monitor during Apollo. Mesh rolls are about 1-inch across, contained in a Teflon film bag..

Table 1. Impurities in Class C nitrogen

Impurity	Specification, ppm	Analysis, ppm
O	10	0.1
Ar	20	9.33
CO2	10	0.1
CO2	10	0.1
H	10	0.1
total hydrocarbon	1	0.1
water	10	0.185

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